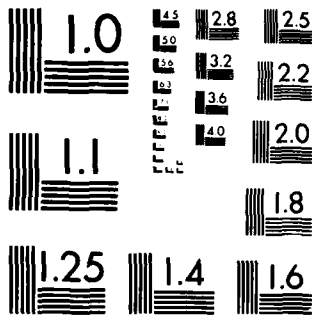END

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MEMORANDUM No. 3733

ROYAL SIGNALS & RADAR ESTABLISHMENT

USE OF ALTERNATIVE SOLVENTS FOR THE LOW — TEMPERATURE LPE GROWTH OF CdTe FILMS

Authors: M Astles, G Blackmore, N Gordon
and D R Wight

PROCUREMENT EXECUTIVE,
MINISTRY OF DEFENCE,
RSRE MALVERN,
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ROYAL SIGNALS AND RADAR ESTABLISHMENT

Memorandum   3733

TITLE: USE OF ALTERNATIVE SOLVENTS FOR LOW-TEMPERATURE GROWTH OF CdTe FILMS

AUTHORS: M Astles,   G Blackmore,   N Gordon and D R Wight

DATE: August 1984

SUMMARY

The use of alternative solvents, in particular Bi and In, has been investigated
for the LPE growth of CdTe thin films at temperatures down to 250°C. In
comparison with earlier work using Te as the solvent, this approach has several
benefits:

   (i)   improved surface topography
   (ii) more complete solution removal
  (iii) more abrupt interfaces
   (iv) improved electrical properties.

SIMS measurements showed that the layers grown from Bi solutions contained
~ 0.1 ppma Bi, while those grown from In solutions contained ~ 100 ppma In.
Doping experiments using In, Sb and As were performed using Bi as the solvent,
and estimated values for the segregation coefficients for growth at 500-400°C
were $k_{In} \sim 6 \times 10^{-4}$, $k_{Sb} \sim 10^{-4}$ and $k_{As} \sim 2 \times 10^{-3}$.

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CONTENTS

1 Introduction

The II-VI compound CdTe has been the subject of much recent research interest since it has several important applications

  (i)   as a substrate for epitaxial growth of (CdHg)Te alloys.
  (ii) as a constituent epitaxial layer in (CdHg)Te/CdTe heterojuctions
  (iii) in solar-energy applications
  (iv) for use in γ-ray detectors.

The crystal growth of device quality CdTe in bulk form has proved to be difficult. In the case of bulk material, the very low thermal conductivity leads to problems in controlling the solid-melt interface shape and hence in producing single crystal material. The retrograde solid solubility of Te in CdTe leads to Te precipitation in bulk CdTe when it is cooled from the growth temperature to room temperature.

These severe problems have led to an interest in a variety of low temperature techniques for growing CdTe eg by (i) MO-VPE[1], (ii) MBE[2,3], (iii) LPE[4], (iv) laser-assisted deposition[5], (v) hot-wall vapour epitaxy[6], and (vi) cathodic electrochemical deposition[7]. The attraction of low growth temperatures is the minimisation of tellurium precipitation, the reduction in diffusion of both matrix and impurity species which is a common difficulty in II-VI compounds, and improved crystallinity.

Recent work on LPE growth of CdTe from Te-rich growth solutions at $\sim$500°C [4] has shown that although good electrical and luminescent properties could be obtained in undoped material, there were problems in producing In or Al doped n-type layers with good electrical properties. This was thought to be due to the loss of Cd or Te from the surface of the grown epitaxial layer during the cool-down to room temperature leading to a high concentration of vacancies (V_{Cd} or V_{Te}) which could then form a deep-level complex such as (V_{Cd}-In_{Cd}) with the In donors. Since this work, the authors have obtained further supporting evidence from SIMS analysis for this mechanism, which will be published at a later date. In order to overcome these difficulties, it was decided to investigate the growth of CdTe at lower temperatures. This entailed investigating the use of alternative solvents to Te since the freezing point of Te is 450°C which sets this as a lower temperature limit for LPE growth from Te-rich solutions. The work described here is concerned mainly with the use of Bi and In solvents, although Sn and Tl were investigated. Details of the LPE growth will be given, together with a description of the assessment techniques used on the layers and the results of these measurements.

2 LPE growth of CdTe using alternative solvents

2.1 Factors affecting choice of solvent

The LPE growth process takes place by inducing supersaturation in an initially single phase solution of the material to be growth (solute) in a suitable solvent. A typical example is the epitaxial growth from a dilute solution of GaAs in Ga. It is desirable, though not essential, to use as the solvent one of the constituent atoms of the material to be grown (in this example, Ga) as this avoids the introduction of foreign atoms into the lattice. In the case of the growth of CdTe, this means that Cd or Te would be suitable solvents. However, as the use of Cd

would involve high partial pressures of Cd (P_{Cd}), the use of tellurium is more desirable and has been widely used as a solvent for LPE growth of CdTe and (CdHg)Te [8,9]. However, as will be mentioned below, tellurium has some properties which make it a poor solvent for LPE growth.

In Table 1, a list of desirable properties and factors involved in the choice of an LPE solvent have been listed, together with the practical effects that these various factors have on the growth process. In Table 2, the values of some of these parameters are compared for several potential solvent materials which have been investigated in this work. It can be seen that Te has several shortcomings, most important being its relatively high melting point and low surface tension. The high melting point means that epitaxial layers can only be grown at temperatures above 450°C.

Table 1   Desirable properties of an LPE solvent

Factor/Property	Effect of the factor
1. High Solubility of the solute in the solvent at low temperatures	High epitaxial growth rates allowing low growth temperatures to be used for a particular layer thickness.
2. Low melting point.	The melting point sets the lowest temperature at which LPE growth can take place.
3. Low vapour pressure.	Little loss by evaporation during the (open-tube) growth process.
4. High purity	Low incorporation of foreign impurities into the growing layer.
5. Low distribution coefficient	Low incorporation of solvent atoms into the growing layer.
6. High surface tension	Facilitates solution removal at end of LPE growth cycle.
7. Low toxicity	Safe handling during loading and unloading.

Table 2   Comparison of Properties of Te, Bi, In and Sn as LPE solvents

		Te	Bi	In	Sn	Tl
1.	Solubility of CdTe at 500°C (mole fraction)	$0.025^{[10]}$	$0.005^{[11]}$	$0.032^{[12]}$	$0.005^{[13]}$	$0.042^{[12]}$
2.	Melting point (°C)	449.5	271.3	156.6	231.97	303.5
3.	Vapour pressure at 500°C (atm)	10^{-3}	10^{-7}	10^{-11}	10^{-13}	–
4.	Purity (available commercially)	69's	69's	79's	69's	59's
5.	Distribution coefficient at 500°C.	–	$\ll 10^{-7}$	6×10^{-4}	10^{-4}	high
6.	Surface tension (dyne cm^{-1}) at 500°C	170	380	530	540	450
7.	Toxicity	high	low	med	low	high

There is much to be gained by being able to conduct the growth at lower temperatures. The low surface tension of Te causes severe problems with solution removal at the end of the growth period, and small droplets of solution are left on the layer surface. These are impossible to remove chemically and cause severe problems in subsequent processing of the wafer apart from the possibility of damage to the grown layer due to differential contraction during the cool-down to room temperature. Although the other solvent materials may possibly introduce impurities into the growing layers, the distribution coefficients, based on SIMS analysis of grown layers are often low enough (eg in the case of Bi) that in practice no problem arises. In the following section, crystal growth work carried out using these solvents (mainly Bi) will be described with the emphasis on the realisation of the potential benefits of the alternative solvent materials.

2.2 LPE growth of CdTe from Bi, In Sn and Tl solutions

The LPE growth work was carried out in a horizontal graphite sliding-boat system illustrated in figure 1. The growth solution and the substrate on which the layer was deposited were held in the high-purity graphite in a single-ended silica work tube under high-purity H_2. The furnace

could be slid on and off the work tube as required. This allowed the
boat to be heated and cooled rapidly, reducing the overall time of the
growth cycle to about 2½ hours. When the boat had been loaded in the
furnace tube, the system was flushed with dry N_2 and Pd-diffused H_2
to a moisture level of ~1ppm. The growth experiment was then begun by
sliding the furnace into position. The boat was heated to a temperature
~10°C above the anticipated liquidus (liquid-solid equilibrium) temperature
for the solution used to allow homogenisation. A temperature ramp of
(typically) 1°C min⁻¹ was then initiated. When the boat had cooled
to a selected temperature normally about 5°C below the liquidus tempera-
ture, the slider in the boat was moved to bring the solution and substrate
into registration. The boat was cooled over a temperature range to grow
the desired layer thickness of ~ 20-30 minutes. The materials used were
99.9999% Te (Cominco), 99.995% Bi (MCP), 99.9999% In (Johnson-Matthey),
99.9999% Sn (Cominco) and 99.99% Tl while the CdTe source material was
polycrystalline undoped CdTe (MCP). The substrates were (111) orientated,
undoped, CdTe single crystal wafers (MCP), and these were polished with
Syton/H_2O_2 and finally etched immediately prior to loading with 2%
V/V bromine/methanol solution to remove ~20 μm from the growth face.

The boat was made from high purity graphite (Poco DFP-2 grade)
and was thoroughly baked in H_2 at 800°C for 5 hours before each run. The
data used to calculate the solution compositions for the various solvents
and growth temperatures is shown in figure 2, gathered from the experi-
mental data of several workers [10-13]. The data for Bi has been
extrapolated into the region of interest from higher temperature
measurements. A small excess of CdTe was generally added to the solutions
to compensate for any errors in the solubility data.

Most of the work used Bi as the solvent and examined the doping of
the layers with In, Sb and As. The dopants were added in elemental form
using 99.9999% In (Johnson-Matthey), 99.9999% Sb (MCP) and 99.9999% As
(Kawecki). In the case of In, a range of atomic fractions from 0 to 1
was investigated, blurring the distiction between solvent and dopant.
Several layers were grown using In as the solvent and in this case,
growth temperatures as low as 250°C were used, among the lowest ever used
for LPE growth of any material. A summary of the growth conditions used
for the various solvents and dopants is given in Table 3.

Table 3   Summary of Growth Conditions

SOLVENT	DOPANT	ATOM FRACTIONS OF "DOPANT"	GROWTH TEMP (°C)	COOL RATE (°C min⁻¹)
Bi	In	0-0.5	500-400	1.0
	Sb	0-0.05	"	1.0
	As	0-0.10	"	1.0
In	~	-	450-400	1.0
			350-300	1.0
			300-250	0.75
Sn	~	-	580-530	1.0
Tl	~	-	460-450	0.5

2.3 Epitaxial layer assessment

2.3.1 Optical microscopy

The layer surfaces were routinely examined under the optical microscope to obtain information on surface flatness, the presence of solution residues and layer thickness from step-height measurements wherein the microscope was focussed first onto the surface of the grown layer and then onto an adjacent ungrown region where the original substrate surface was visible. The difference in vertical focussing position gave an estimate of layer thickness accurate to ± 1.5 μm. This procedure was used since no satisfactory delineation etch could be found to reveal the interface position on a cleaved edge.

2.3.2 Capacitance - voltage measurements

Because of the difficulties of making reproducible ohmic contacts to low conductivity CdTe, capacitance - voltage measurements on Schottky barriers have been used to assess the carrier concentrations of the epitaxial layers. Evaporated Al was used to form the Schottky barriers, with an array of small area contacts and a large area contact being deposited. The capacitance was measured between one small contact and the large area contact, using a computer-controlled Hewlett Packard 4275 LCR meter. This enabled a large number of readings to be recorded, averaged and corrected for series resistance and then plotted in a short time. The usual $1/C^2$ vs V graph was plotted and then differentiated to give a carrier concentration profile against depth. By using an array of small Schottky barrier contacts, it was possible to measure the uniformity of carrier concentration across a layer. In some cases, the layers were etched to remove 4-5 μm and a new set of Al contacts evaporated to obtain a carrier concentration profile deeper into the layer.

2.3.3 "Water-drop" measurements

This technique for measuring minority carrier diffusion lengths was first applied to GaAs by Allenson and Bass [14] and its applicability to CdTe has recently been described by Wight et al [15]. It basically involves measuring the photovoltage produced by illuminating a Schottky barrier produced on the sample surface by a small drop of dilute (0.01N) Sulphuric acid. The photovoltage is measured as a function of wavelength between 600 and 900nm. Data on the absorption coefficient α in this wave length range is required and was obtained by direct measurement on thinned samples of bulk CdTe. A plot of quantum yield (arb-units) vs. absorption coefficient (cm^{-1}) is then produced which can then be computer fitted to a theoretical expression using adjustable values of diffusion length L and surface recombination velocity S.

2.3.4 Secondary - Ion Mass Spectrometry (SIMS)

The use of SIMS for the quantitative measurement of impurity concentrations in epitaxial layers as a function of depth is an invaluable aid in crystal growth research.[16] Several of the layers

in this work have been assessed using a CAMECA IMS 3F machine which uses a 12.5KV beam of O_2^+ primary ions focussed to 10-20 μm diameter rastered over an area of 500 μm x 500 μm giving an erosion rate for CdTe of about 3 μm hr^{-1}. The secondary ions from the central 60 μm diameter were analysed in a similar way to that used in a conventional mass spectrometer. Many impurity elements have been calibrated for CdTe and (CdHg)Te by the use of standards which have been analysed by spark-source mass spectrometry and atomic absorption spectroscopy. (17)

3. Results

3.1 LPE layer topography

As expected, the wipe-off of growth solution when using In and Bi solutions was very much improved in comparison with Te and in most cases was ~ 100% as can be seen in figure 3. It was also found that the layer surface topography was very much better with In and Bi solutions as can be seen in figure 4, which shows a comparison of the as-grown surfaces obtained from Te- and Bi-solution growth. The surfaces produced by Sn- and Tl-solution growths were generally poor (figure 5) and in the case of Tl were so poor that very little solution removal was achieved. Layer thicknesses were measured using the step-height technique mentioned earlier. Typical average growth rates were 0.05 μm $^\circ$C^{-1} for Bi solutions (500-400°C) and 0.16 μm $^\circ$C^{-1} for In solutions (300-250°C).

3.2 Bi solution growth results

Typical capacitance/voltage profiles for layers growth from Bi solution are shown in figure 6. The layer grown at 500-400°C had a carrier concentration of p~8 x 10^{16}cm^{-3} (fig 6a) while a layer grown from 400-300°C had a graded carrier concentration from p=4 x 10^{14}cm^{-3} near the surface to p=2.6 x 10^{15}cm^{-3} at 3 μm depth (figure 6b). The concentration of Bi in the layers measured by SIMS was ~ 10^{15}cm^{-3}. It is possible that the p-type background level was due to some other impurity in the Bi or to a native defect. However, in later runs, where a higher purity Bi source (99.9999%) was used, the carrier concentrations obtained were very similar to those mentioned above.

The effect of doping with In was investigated over the range X_{In} = 2 x 10^{-2} to 0.5 and the results are shown in figure 7 in a plot of log ($N_d - N_a$) vs log at.fr.In, the slope of the line being ~ 1. Figure 8 shows a SIMS profile of one of these In-doped layers showing a very uniform incorporation at the ~ 5ppm level. The distribution coefficient k_{In} calculated from these SIMS analyses in ~ 6 x 10^{-4}.

A series of runs growing from Bi + added In dopant was performed to investigate the variation of minority carrier diffusion length as measured by the "water-drop" technique with carrier concentration, the In atom fraction being varied from 0 to 1. The results are given in Table 4.

Table 4   Minority carrier diffusion length measurements

T_G = 500-400°C at 1°C min^{-1} cool rate, Bi/In solutions.

atom fraction indium X_{In}	carrier concentration (type) (cm^{-3})	diffusion length (μm) L_e or L_h
0	9 x 10^{16} (p)	2 (L_e)
0.1	2 x 10^{16} (n)	0.5 (L_h)
0.2	3 x 10^{16} (n)	1.0 (L_h)
0.5	6.9 x 10^{16} (n)	0.25 (L_h)
1.0*	1.5 x 10^{17} (n)	30 (L_h)

* T_G = 450-400°C at 1°C min^{-1}

The low values of L_h for the layers grown from mixed Bi + In solutions
may be due to the presence of impurities such as Li, or Na which were
found in the SIMS analysis of some layers at the 1-10ppm level. It is
interesting to note that the minority carrier diffusion length
increases dramatically when pure In is used as the solvent, and the
LPE layer in this case was found to be free of Li or Na. The effect
of specific impurities on minority carrier transport properties obviously
requires further investigation.

Doping experiments were also performed using Sb and As to
obtain p-type material. Both dopants were found by SIMS and C/V
measurements to be uniformly distributed through the layer with good
agreement between the carrier concentration and the chemical analysis
results implying that these species were ∿ 100% active in the layers. For
example, figure 9 shows a SIMS profile of an As-doped layer, showing a
fairly constant level of ∿ 3 x 10^{18}cm^{-3} As in the layer and an abrupt
interface with the substrate. The C/V measurement of this layer gave a
carrier concentration of p=2.8 x 10^{18}cm^{-3} in good agreement with the SIMS
result. Approximate distribution coefficients for Sb and As from these
results have been calculated as k_{Sb}∿10^{-4}, k_{As} ∿ 2 x 10^{-3}.

3.3 In solution growth results

Several layers were grown from pure In solutions at a variety of
temperatures. The SIMS analyses showed that In was incorporated in the
layers at the 10^{18}cm^{-3} level fairly independent of growth temperature.
Figure 10 is a typical SIMS profile for In on an epitaxial CdTe layer grown

from an In solution at 300–250°C, showing an abrupt interface with the substrate characteristic of these low temperature growths. In this case, there is a fall in the In signal of two orders of magnitude in $\sim$ 0.1μm. The electrical activity measured by C/V profiles on these In solution grown layers did vary with growth temperature although the minority carrier diffusion lengths were always in the range 30–40 μm. For a layer grown at 450–400°C, the carrier concentration was n $\sim$ 1.4 x $10^{17}cm^{-3}$ while a layer grown at the lower temperature of 300–250°C was n $\sim$ 3.5 x $10^{17}cm^{-3}$, indicating that more of the In dopant is electrically active when the growth temperature is reduced. This effect was also illustrated by a experiment whereby a piece of one of the low temperature In solution grown layers was given a 10 minute heat treatment at $\sim$ 500°C in the LPE growth system. After this treatment the carrier density was dramatically reduced to n $<10^{14}cm^{-3}$. This result is similar to that obtained in earlier work[4] in which In doped CdTe was grown by Te-rich LPE at $\sim$ 500°C. These new results support the earlier contention that the low electrical activity seen in the In-doped layers grown at higher temperatures ($\sim$ 500°C) was due to a post-growth annealing involving loss of Cd from the layer surface[4].

3.4 Sn and Tl solution results

A few layers were grown from Sn and Tl solutions. Generally, the results were not encouraging. In both cases, the surface topograph the layers was poor as shown earlier in figure 5. With Sn solutic   .ne wipe-off of growth solution was good, and C/V measurements showed _ne layer to be p $\sim$ 1 x $10^{17}cm^{-3}$ although the minority carrier diffusion ^{1}e th was <0.1 μm. For Tl solutions, it appeared that the Tl incorporation in the layer was so high that an alloy of Tl_2Te_3-CdTe was being grown, as evidenced by marked changes in lattice parameter and by a distinct colour change at the interface between the CdTe substrate and the grown layer.

These solvents, therefore, do not appear promising for the growth of high quality CdTe epitaxial layers.

4. Discussion

The work reported here shows the great improvements in materials properties of CdTe that can be obtained by performing LPE growth at lower temperatures. If the right solvents and growth rates are used, the surface topography can be mirror smooth. The problems associated with higher temperature growth, in particular (i) inter-diffusion at interfaces and (ii) poor electrical properties, can be overcome by this technique, enabling the mechanisms involved in diffusion and impurity compensation to be studied.

Although the number of possible solvents for low temperature LPE growth is limited by considerations of

    (i) solubility
    (ii) inertness
    (iii) incorporation into the layer

there are obviously merits in this approach for specific materials and device applications. Also, the area of mixed solvent growth deserves exploration since it is possible that the incorporation of one solvent species could be affected by the presence of the other.

The use of low temperature LPE growth should provide a way of combining the good minority carrier and luminescent properties of LPE material with the good surface topography and abrupt interfaces characteristic of MBE and MO-VPE growth.

5. Conclusions

The use of alternative solvents, principally Bi and In, for the low temperature LPE growth of CdTe down to 250°C has been described. Several benefits of this approach compared to the use of Te-rich solutions have been found —

   (i)   better surface morphology
   (ii) improved solution removal
  (iii) abrupt interfaces
   (iv) improved electrical properties

Typical electrical results for Bi solution growths are $p \sim 9 \times 10^{16} cm^{-3}$ at room temperature and a minority carrier diffusion length $L_e \sim 2$ µm, while for In solution growths, $n \sim 10^{17} cm^{-3}$ with a minority carrier diffusion length L_p > 30 µm. The use of In, Sb and As as n- and p-type dopants has been explored for Bi-solution growth, the estimated distribution coefficients being $k_{In} \sim 6 \times 10^{-4}$, $k_{Sb} \sim 10^{-4}$, $k_{As} \sim 2 \times 10^{-3}$.

Although Tl and Sn were investigated as possible solvents, the results for these were not encouraging and further work on these was not carried out.

Acknowledgement

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    K J Timmins, G W Blackmore,   J Electronic Materials 12(1983)879
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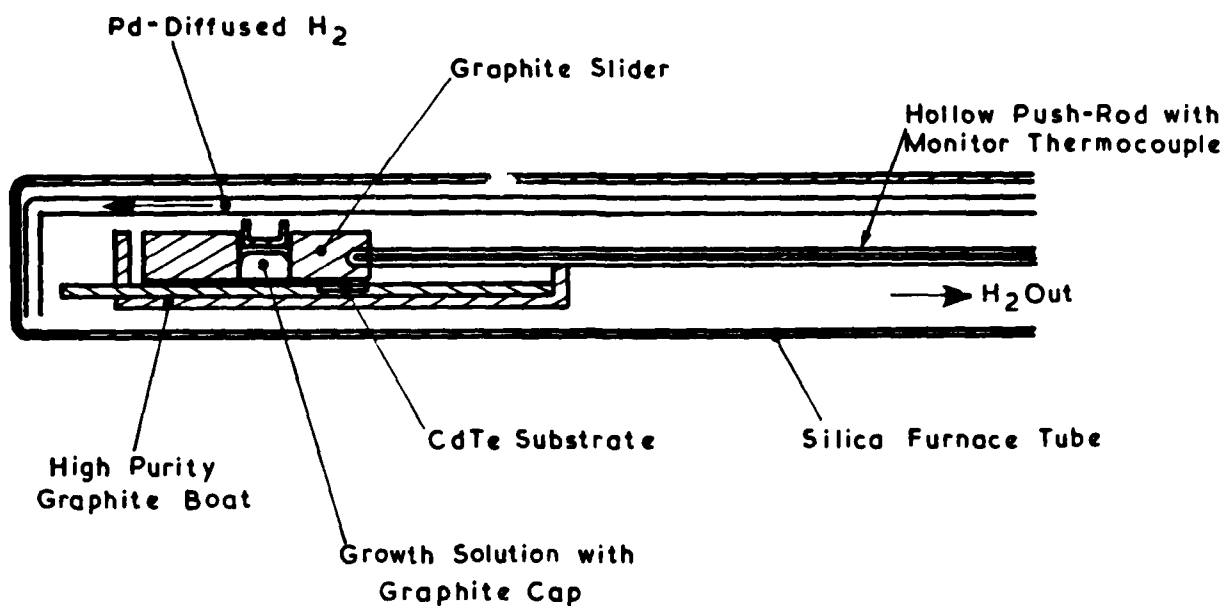
Figure 1   Schematic of LPE growth system

SOURCES   In TAI & HORI (12)
          Te LORENZ   (10)
          Bi RUBENSTEIN (11)
          Tℓ TAI & HORI (12)
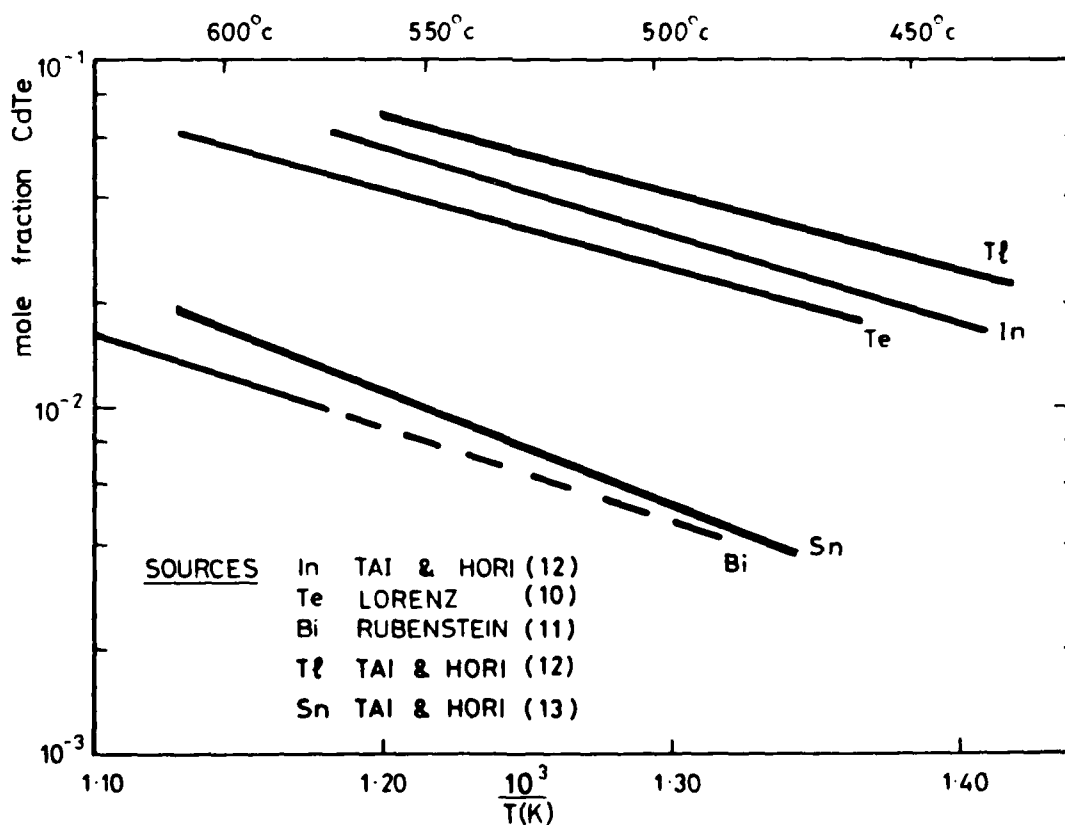   Sn TAI & HORI (13)

Figure 2   Solubility data for CdTe in In[12], Te[10], Bi[11], Tℓ[12] and Sn[13] as a function of 1/T(K)
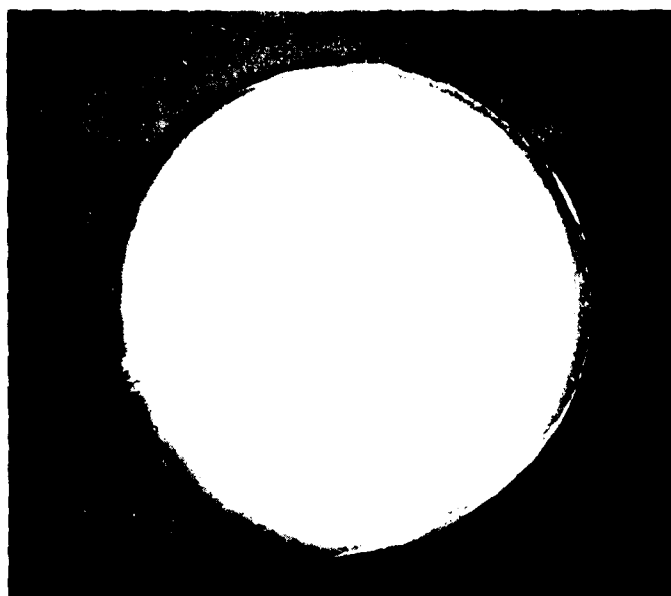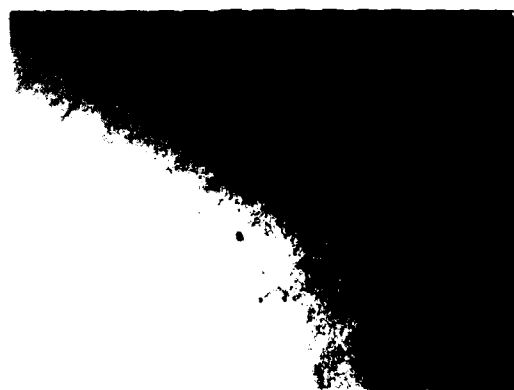
Figure 3   Complete wafer of CdTe grown from Bi solution showing
                complete removal of solution residues

Te   Solution   ⊢————⊣   Bi   Solution
                                  100 μm
(111)B   (111)B

Figure 4   Surface topography of LPE layers of CdTe grown from Te
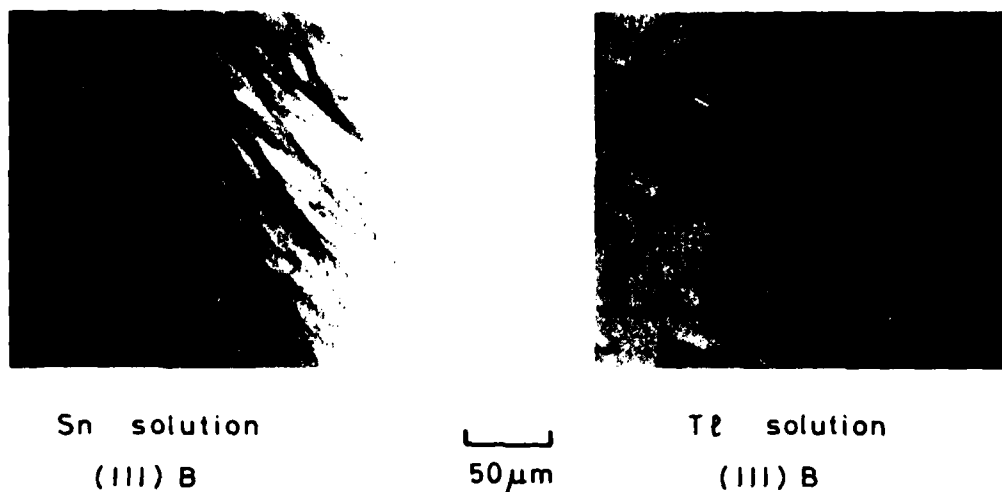   and Bi solutions onto (111)B CdTe.

Sn solution
(III) B

50 μm

Tℓ solution
(III) B

Figure 5   Surface topography of LPE layers grown from Sn and
Tℓ solutions onto (III)B CdTe.

(a)

CdTe : Bi
500 - 400°C

$p\,(cm^{-3})$

10^{17}

10^{16}

0   0.1   0.2   0.3

DEPTH (μm)

(b)

CdTe : Bi
400 - 300°C

$p\,(cm^{-3})$

10^{15}

10^{14}

0   1.0   2.0   3.0

DEPTH (μm)
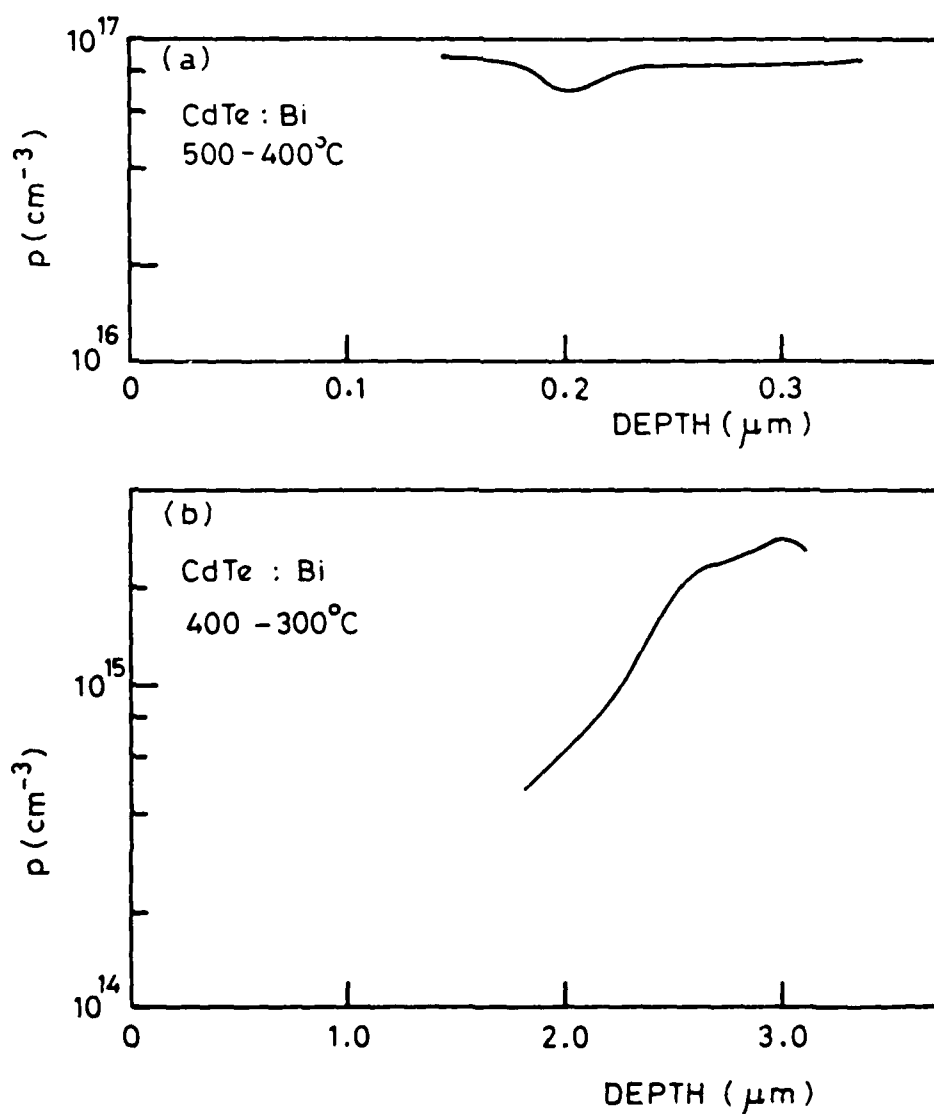
Figure 6   Capacitance/voltage profiles of (N_a-N_d) against depth
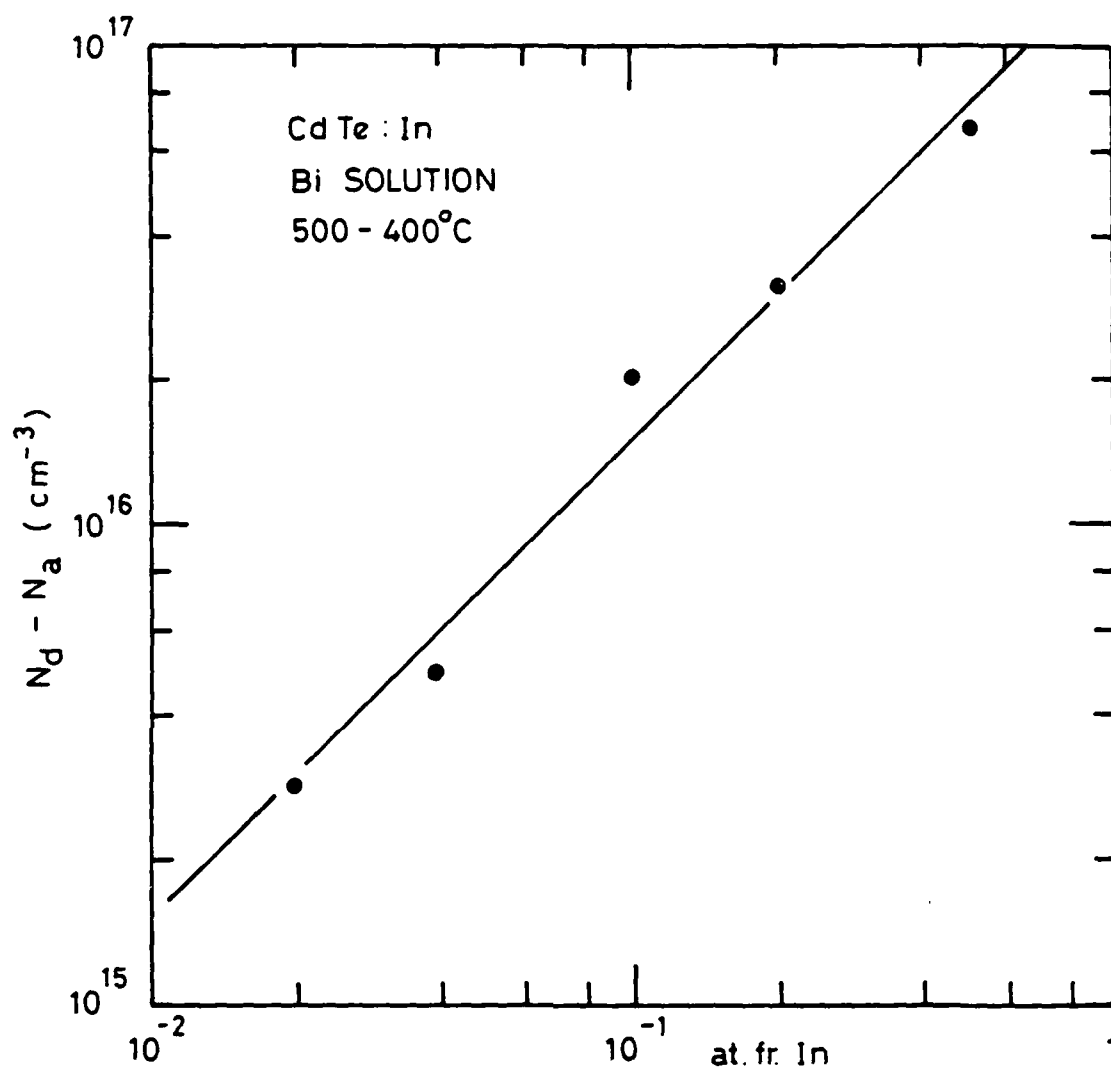
Figure 7   Carrier concentration (N_d-N_a) (cm^{-3}) as a function of atom
                fraction In for growth of CdTe from Bi solutions at 500-400°C
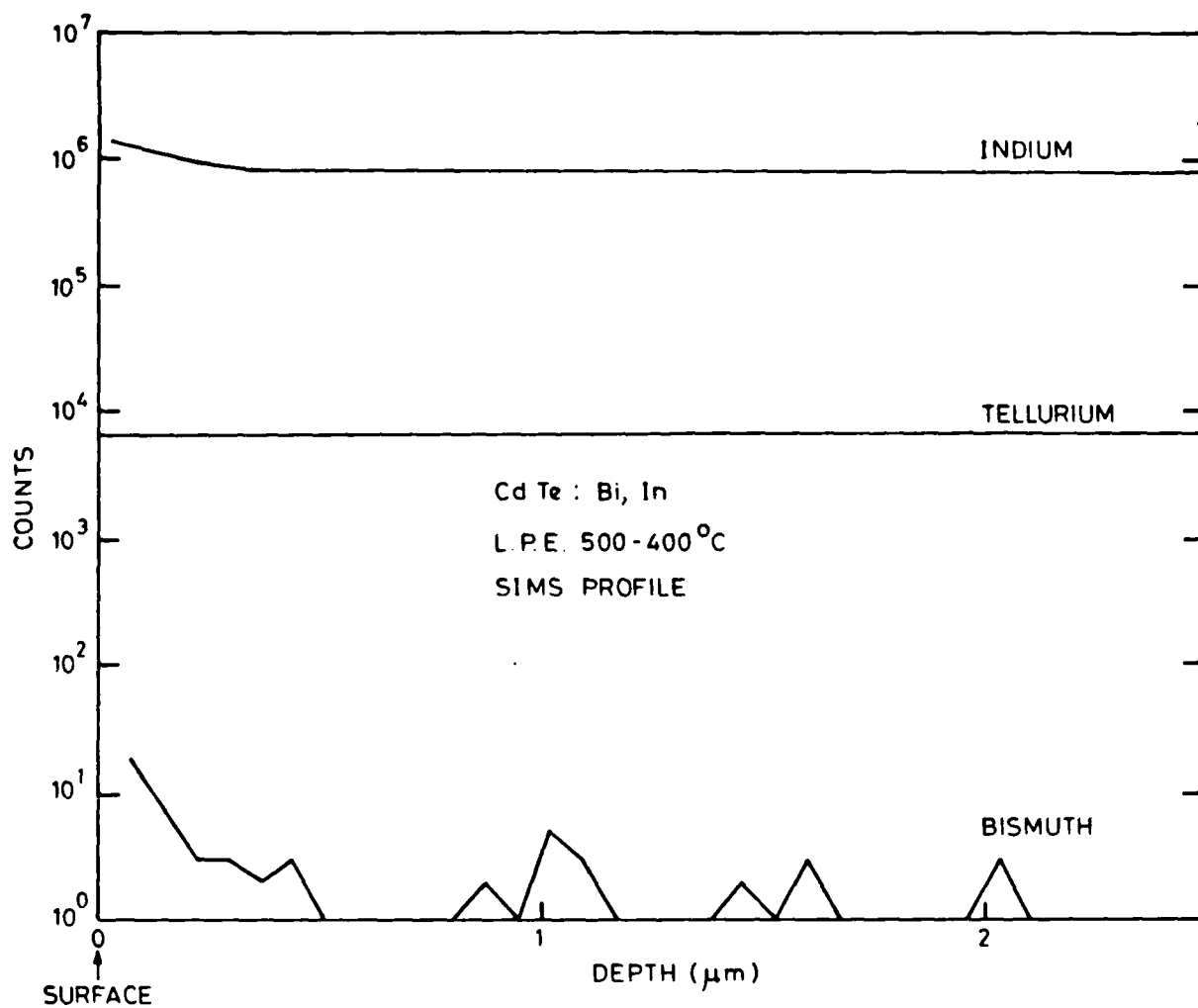
Figure 8   SIMS profiles of In, Te and Bi in an In-doped CdTe layer grown from Bi solution at 500-400°C.
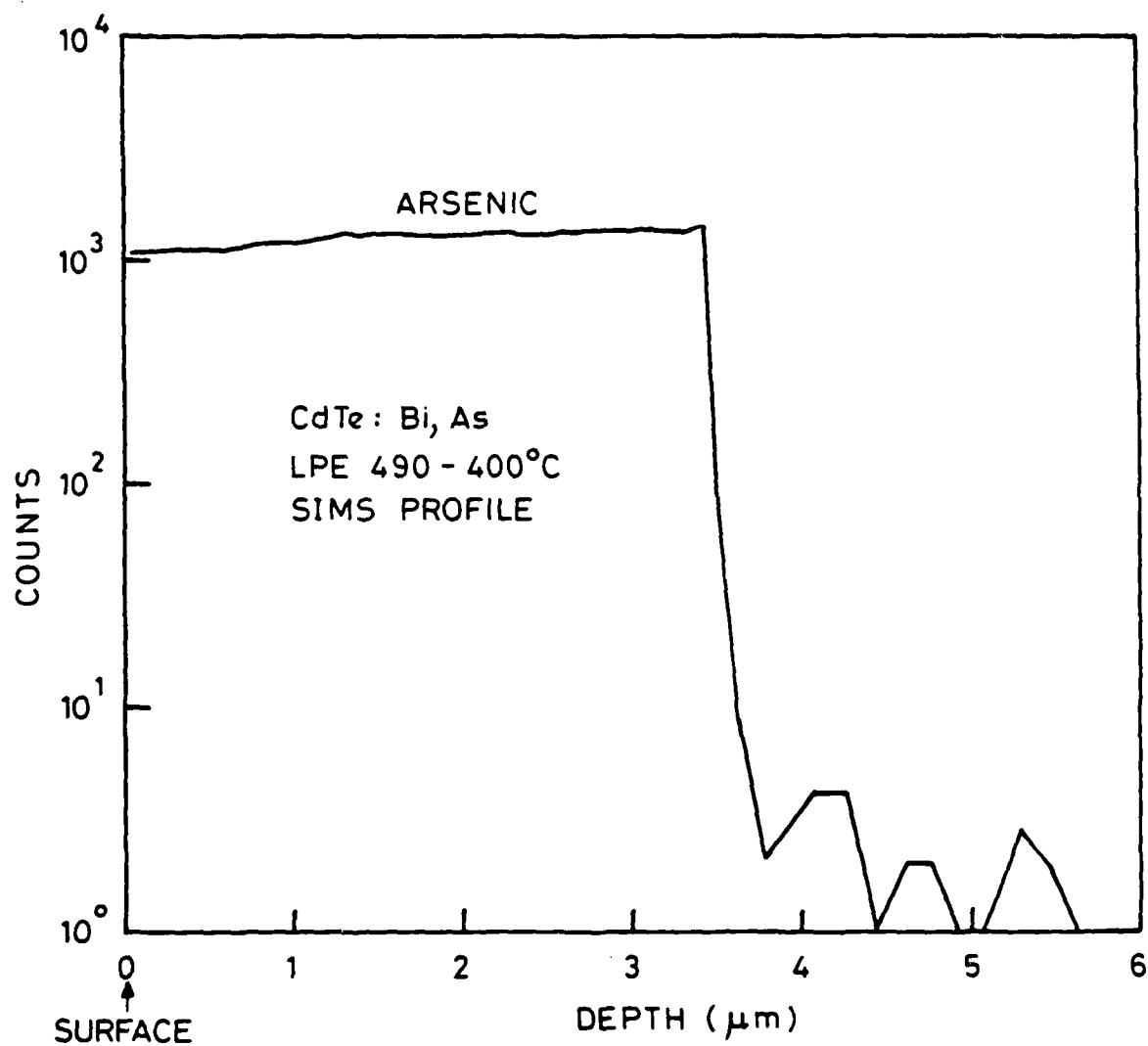
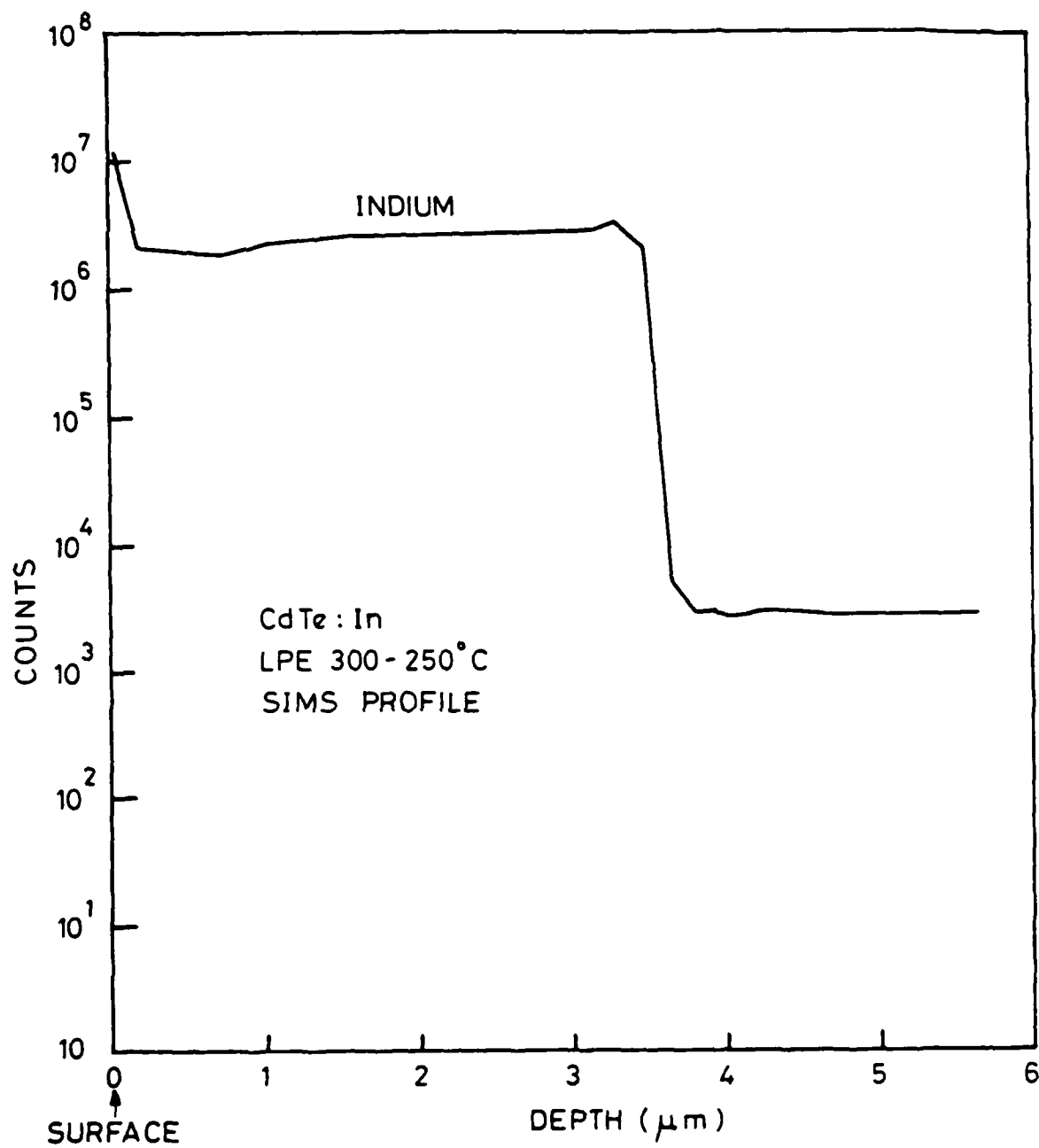Figure 9   SIMS profile of an As-doped CdTe layer grown from Bi solution at 500-400°C.

Figure 10   SIMS profile of an LPE layer of CdTe grown from In solution at 300-250°C.

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